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DOMESTIC PREPAREDNESS PROGRAM: TESTING OF PHOTOVAC MicroFID HANDHELD FLAME IONIZATION DETECTORS AGAINST CHEMICAL WARFARE AGENTS

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13. ABSTRACT *(Maximum 200 words)*

The commercially available Photovac MicroFID Handheld Flame Ionization Detector was tested against HD, GB, and GA vapor at various conditions. This report characterizes the chemical warfare (CW) agent detection potential of these detectors. It is intended to provide the emergency responders concerned with CW agent detection an overview of these detectors' characteristics based on results of the evaluation.

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PREFACE

The work described herein was authorized under the Expert Assistance (Equipment Test) Program for the U.S. Army Soldier and Biological Chemical Command (SBCCOM) Program Director for Domestic Preparedness. This work was started in December 1998 and completed in January 1999.

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DOMESTIC PREPAREDNESS PROGRAM: TESTING OF PHOTOVAC MicroFID HANDHELD FLAME IONIZATION DETECTORS AGAINST CHEMICAL WARFARE AGENTS

1. INTRODUCTION

In 1996, responding to Public Law 104 - 201, the Department of Defense (DOD) formed the Domestic Preparedness (DP) Program. One of the objectives is to enhance federal, state and local capabilities to respond to Nuclear, Biological and Chemical (NBC) terrorism incidents. Emergency responders who encounter a contaminated or potentially contaminated area must survey the area for the presence of toxic or explosive vapors. Presently, the vapor detectors commonly used are not designed to detect and identify chemical warfare (CW) agents. Little data are available concerning the ability of these commonly used commercially available detection devices to detect CW agents. Under the Domestic Preparedness (DP) Expert Assistance (Test Equipment) Program, the U.S. Army Soldier and Biological Chemical Command (SBCCOM) established a program to address this need. The Design Evaluation Laboratory (DEL) at Aberdeen Proving Ground, Edgewood, Maryland, performed the detector testing.

2. OBJECTIVE

The objective of this testing program is to screen different detection devices currently being used by first responder organizations for their ability to detect chemical warfare agent vapors. The detectors selected for testing are based on a Battelle Memorial Institute (BMI) survey[1] that identifies the detectors most likely to be used by the local responders in the Baltimore vicinity in the event of a terrorist incident involving CW agent(s). The approach is to seek the concentration level where a repeatable detection reading is produced for the respective agent. It was impractical to cover every conceivable agent and condition. Therefore, tests were planned to include a range of temperature, humidity, and concentration effects for representative CW agents. This evaluation includes measurement of CW agent detection performance for one of the selected Flame Ionization Detectors (FIDs), the MicroFID. The nerve agents used for testing included Tabun (GA) and Sarin (GB), and the blister agent used was Mustard (HD). These were chosen as representative CW agents because they are believed to be the most likely threats and have been deployed on several occasions. Each observation was confirmed with repeated trials. The original Domestic Preparedness Test Plan is attached in the Appendix. Modifications were made to the test plan as necessary following the basic guidelines for testing of the individual detectors.

[1] Battelle Memorial Institute, CBIAC Task 252, Effect of Emergency Responder Chemical Detection Equipment for the Detection of Chemical Warfare Agents Study, 17 July 1997.

3. EQUIPMENT AND TEST PROCEDURES

3.1 DETECTOR DESCRIPTION

The Photovac MicroFID Handheld Flame Ionization Detector is manufactured by Perkin-Elmer Corporation, Photovac Monitoring Instruments, PE Photovac. The devices tested were new and evaluated in the "as received" condition. No attempt was made to optimize their chemical agent detection capability. No pre-test theoretical assessment was made on any of the items except to learn the operating procedures prescribed in the User's Manuals[2] which were provided with the detectors. Detailed operation information and a technical description of the detectors are found in the referenced User's Manuals. Conclusions are based solely on the results of the detector responses to CW challenges during this testing. No other aspects of the test items were investigated.

Figure 1 is a digital photograph of a MicroFID. Three MicroFIDs were purchased and randomly labeled A, B, and C. The detectors were operated with the battery charger connected to 110 V AC to ensure optimum battery condition during testing. The built in hydrogen cylinder of each detector was filled as needed with ultra high purity hydrogen to provide a consistent flame. Inlet filters were changed regularly, the flame was ignited, and the parameters were set according to the user's instruction manual.

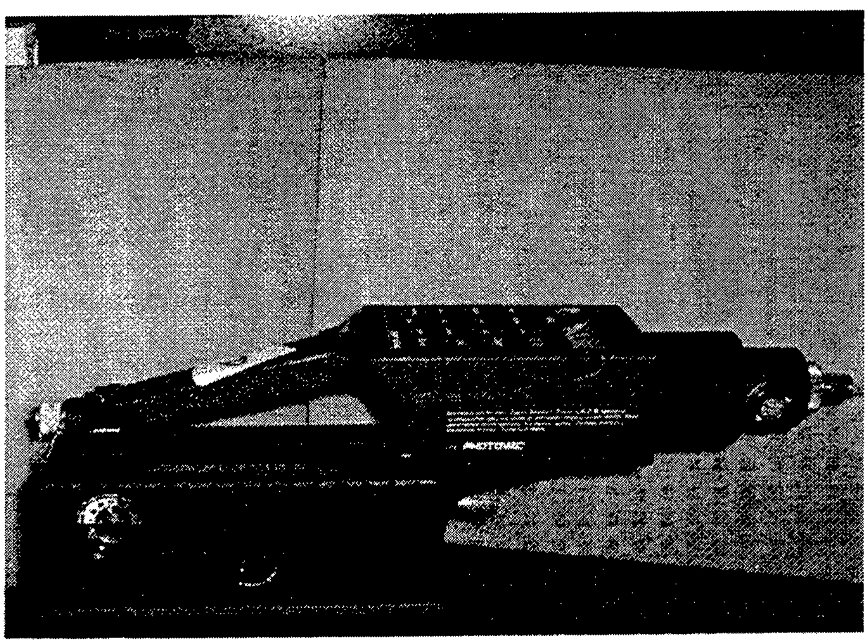

Figure 1: MicroFID

[2] User's Manual, MicroFID Flame Ionization Detector, PE Photovac, Markham, Ontario L3R 8E5, Canada, July 1997.

3.2 CALIBRATION

Each detector was allowed to stabilize before initiating the calibration procedures. Calibrations were performed daily per instructions provided with the detector. Calibration allows the detector to display concentration in parts per million (PPM) units equivalent to a calibration gas. The calibration procedure requires adjusting the detector baseline zero point by challenging with 'zero air' ($\leq$ 0.1 PPM total hydrocarbons). Then the detector is challenged with a calibration 'span gas' of known concentration to set the sensitivity. Mylar sample bags were filled with the appropriate gas and used to challenge the detectors. Analyzed +/-2% methane gas at 499.7 PPM in hydrocarbon free air was recommended and used as the span gas for these evaluations. Methane at 100 PPM concentration was used as a control to confirm the adequacy of the calibration. The detector was re-calibrated if the control methane reading was greater than +/-10% of the expected reading. This confirms that the detector was accurately reading methane even if no response readings were seen during a CW agent challenge or when ambient air readings were greater than the CW agent challenge readings.

3.3 AGENT CHALLENGE

The methodology for agent generation is based on the Multi-Purpose Chemical Agent Vapor Generation System[3] (Vapor Generator). Figure 2 is a flow schematic of the basic agent generator. The agent challenges were conducted with zero air and CASARM grade (High Purity) CW agents. The vapor generator permits preconditioning of a detector with humidity and temperature conditioned air prior to challenging it with similar air containing the CW agent. This corrects for any potential influence from different background temperature and humidity conditions. Occasionally, the detectors were checked with the 100 PPM methane gas after the agent challenge to observe residual effects and/or calibration drift.

Agent testing followed successful detector calibration. First, conditioned air at the desired temperature and humidity from the vapor generator system was sampled by the MicroFID for approximately one minute to establish the stable "background reading" of the detector for the air at each condition. The background reading (baseline) at the testing condition is required to establish the net detector response from the agent challenge reading. The net detector reading is the challenge reading minus the background reading.

Agent challenge begins when the vapor generation system solenoids are energized to switch the air streams from conditioning air only to similarly conditioned air containing the agent. Each detector was tested three times under each condition. The agent challenge time was approximately 5 minutes to allow the maximum detector response. Detector response readings were recorded every minute during the agent challenge. Also, the times for clear down back to the baseline after the agent challenge were noted.

[3] Ong, Kwok Y., Multi-Purpose Chemical Agent Vapor Generation System, ERDEC-TR-424, Aberdeen Proving Ground, MD, July 1997.

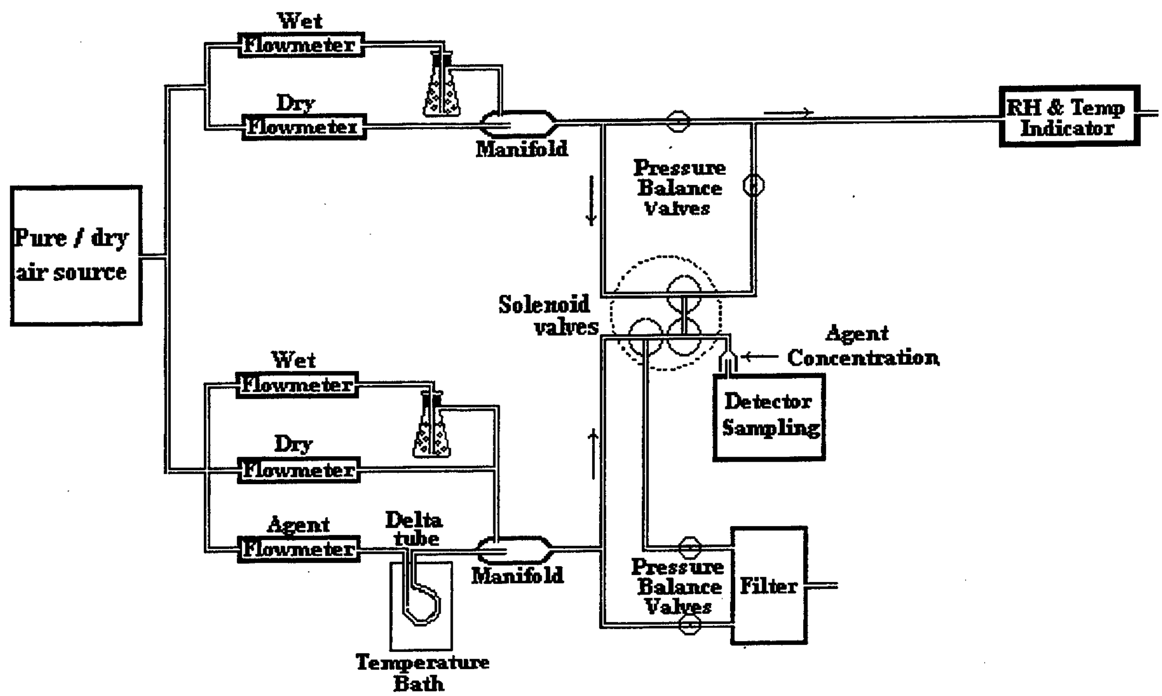

Figure 2: Basic Agent Generator

The MicroFID detectors were each tested with the agents GA, GB and HD at different concentration levels at ambient temperature and 0% relative humidity (measurement tolerance 2%) in an attempt to determine the minimum detectable level (MDL) and establish a response curve. The detectors were also tested at other relative humidity conditions (50% and 90%) to observe humidity effects. The actual ambient temperatures (19-25°C) and humidity values were recorded during testing, and adjustments made as necessary for the actual conditions.

The detector response in PPM was observed and recorded. The response readings are relative to the calibration gas. Therefore, the observed detector reading is in PPM methane equivalent units. Response factor (RF) is an indication of the relative sensitivity of a detector to the concentrations of a compound vapor at each condition compared to the calibration gases used. RF is required by MicroFID (Flame Ionization Detector) users to determine the combustible organic compounds in the sample which are ionized by the flame. The displayed reading shows the total concentration of all ionizable compounds in the sample. Calculated RFs are commonly entered into a detector's memory to enable the instrument to automatically display the correct concentration readings. The RF is calculated by dividing the actual concentration in the test air (in PPM) by the net detector reading (in PPM).

$$\text{Response Factor} = \frac{(\text{Actual Challenge Concentration})}{(\text{Detector Reading}) - (\text{Background Reading})}$$

Hence, methane will have a response factor of 499.7 PPM/(499.7 PPM – 0 PPM) = 1. Ideally, a compound's RF values should be constant for a given detector model. Higher RF values indicate the detector is less sensitive in detecting that compound vapor. In general it is acceptable to use a detector for most non-lethal compounds when its RF values are less than ten. However, for CW agent detection, the RF values must be much lower due to the high toxicity of agents, or casualties will result. Response factors for the CW agents determined during the evaluation were calculated and recorded in the data tables. These data tables include all the recorded data for each test and are located in the DEL Laboratory Notebook 98-0040.

3.4 AGENT VAPOR CONCENTRATION QUANTIFICATION

The generated agent vapor concentrations were recorded in micrograms/liter (ug/l) and PPM units for the RF calculations. Note that ug/l and mg/m^3 (milligrams per cubic meter) are equivalent or interchangeable units for expressing concentration measurements.

The generated agent vapor was quantified by manual sample collection methodology[4] using the Miniature Continuous Air Monitoring System (MINICAMS) manufactured by O. I. Analytical, Inc., Birmingham, Alabama. The MINICAMS is equipped with a flame photometric detector (FPD). This system normally monitors air by collection through sample lines and subsequently adsorbing the CW agent onto the solid sorbent contained in a glass tube referred to as the pre-concentrator tube (PCT). The PCT is located at the MINICAMS inlet where the concentrated sample is heat desorbed into a gas chromatographic capillary column for subsequent separation, identification, and quantification. The sampling method of the MINICAMS was modified to manual sample collection for detector testing. The sample was collected directly into the PCT for the determination of agent concentrations. This eliminates potential loss of sample through sampling lines and the inlet assembly in order to use the MINICAMS as an analytical instrument.

For manual sample collection, the PCT is removed from the MINICAMS during its sample cycle and connected to a measured suction source to draw the vapor sample from the agent generator. The PCT is then re-inserted into the MINICAMS for analysis. The vapor concentration derived is based on the sample volume and the amount of agent found in the sample by comparing to a standard curve of calibrations using known concentrations of agent standards. The calibration of the MINICAMS is performed daily using the appropriate standards for the agent of interest.

4. RESULTS AND DISCUSSION

4.1 MINIMUM DETECTABLE LEVELS

The minimum detectable level (MDL) for the MicroFID detectors A and B are shown in Table 1 for each agent at ambient temperature and zero percent relative humidity (RH). The 0% RH condition was used to establish the MDL because the detectors were zeroed and calibrated using zero (dry) gas. It should be noted that no data results are given for detector C due to its failure to power up on the second day of testing.

[4] Ong, K. et al, Analytical Methodology for Quantitative Determination of VX, ERDEC-TR-476, Mar 98.

The MDL concentrations are expressed in mg/m^3 with equivalent parts per million (PPM) values given in parentheses. The agent vapor concentrations are analyzed in units of mg/m^3, but the detectors display a response in units of PPM which are also needed to calculate the response factors. The values shown are the representative values recorded for detectors A and B. The MDL expressed here represents a detector display response of at least 0.5 PPM. In the table, the concentrations identified in the footnote as 'No response to concentrations up to this level' means that the detector was tested up to the given concentration with no displayed results or with no reproducible response. Some detector responses showed PPM indications less than 0.5 that might be construed as a "detection" but their responses remained less than 0.5 PPM even at the highest agent concentrations tested. Those results are identified in the table with the footnote 'Minimal response to concentrations up to this level'.

Table 1 also includes a column listing the Joint Service Operational Requirements (JSOR) for point detectors. The current Joint Services Operational Requirement (JSOR) for point sampling CW agent detectors is 0.1 mg/m^3 for the G (nerve) agents and 2 mg/m^3 for the H (blister) agents. It is clear from the results that the MicroFID in its current configuration will not detect CW agents at the JSOR levels even under ideal conditions. Although they demonstrated some detection capability toward the CW agent vapor given sufficient concentration, the sensitivities were considered by the authors too low and too non-specific to provide needed warning. A reliable response curve could not be produced during the evaluation for any agent using the MicroFIDs.

Table 1. Minimum Detectable Level (MDL) for HD, GA, and GB at Ambient Temperatures and 0% Relative Humidity

	MicroFID MDL in mg/m^3 (PPM)		
AGENT	Detector A	Detector B	JSOR Requirements
HD	46 (7.03)[a]	23.5 (3.59)[b]	2 (0.3)
GA	13.9 (1.30)	13.9 (1.30)	0.1 (0.017)
GB	27.8 (4.84)	27.8 (4.84)	0.1 (0.017)

[a] No response to concentrations up to this level
[b] Minimal response to concentrations up to this level

4.2 RESPONSE FACTORS

Response factor (RF) values for the CW agents tested were calculated at ambient temperatures (19-25C) and the relative humidity conditions of 0, 50 and 90%. The ranges of calculated RF values at the average temperature and humidity conditions are summarized in Table 2. The RF values represent the results of multiple challenges of both units at agent concentrations between 3 and 60 mg/m^3. Response factors calculated for the CW agents tested are shown in the Data Tables in Appendix B. No direct relationship could be defined between RF and agent concentration.

Ideally, the RF values for both detectors should be very similar. However, these detectors responded to CW agents differently than to the calibration gas, perhaps due partly to the volatility differences, and exhibited varied and unpredictable ranges of RF values. In fact, A and B detectors behaved differently on any given day. For example, MicroFID A would be more sensitive than MicroFID B on one day, then the reverse was observed on another day even when both detectors were repeatedly responding correctly to the calibration gas.

Results consolidated in Table 2 reflect the wide ranges of RFs observed at different concentrations and conditions for the detectors tested. It presents the highest and the lowest RF calculated from results of each tested condition. The highest RFs reported as NR (No Response) mean the detector failed to respond even at concentrations much higher than the JSOR requirements. Essentially, this gives a value of zero for the denominator of the RF equation causing the RF to go to infinity. No consistent response factor for any detector or condition could be determined.

Table 2. Range of Response Factors (RFs) for MicroFID A and B at Various Conditions

Agent	Average Temp., °C	Relative Humidity, %	MicroFID A		MicroFID B	
			Lowest RF	Highest RF	Lowest RF	Highest RF
HD	20	0	6.41	NR*	5.98	NR*
HD	20	50	23.29	93.19	9.32	18.63
HD	20	90	90.1	NR	11.26	15.02
GA	20	0	0.7	NR	1.89	NR
GA	20	50	1.33	1.49	1.95	2.82
GA	20	90	1.0	1.14	0.8	1.33
GB	20	0	4.4	NR	4.03	NR
GB	20	50	2.52	19.08	2.69	8.18
GB	20	90	4.46	NR	4.78	NR

* NR indicates no detector response = division by zero = infinity.

The detectors were checked with the methane gas at 100 PPM after calibration with the 499.7 PPM methane calibration gas. And, frequently after the agent challenges, the detectors were rechecked with the 100 PPM methane gas to observe residual effects and/or calibration drift. The detector responses to these challenges suggest that the detector sensitivity was not degraded by exposure to CW agent vapor. Calculating the response factors on the 100 PPM methane gas challenges recorded during all testing for both detectors shows a RF range of 1.04 to 1.13. In contrast, the RFs recorded during the CW agent tests shown in Table 2 show no consistency between detectors A and B, and an unacceptable range between low and high values for all agents tested. Observations suggest that these detectors cannot be relied upon for CW agent vapor detection and warning.

4.3 RELATIVE HUMIDITY EFFECTS

The MicroFID baseline response did not appear to be affected by relative humidity changes. All baseline readings were zero at the different humidity conditions tested even though the detectors were calibrated with zero (dry) air only. No conclusive RH effects could be determined due to the large ranges and inconsistencies of the detector readings.

5. CONCLUSION

This test evaluation includes measurement of CW agent detection performance using the nerve agents, Tabun (GA) and Sarin (GB), and the blister agent, Mustard (HD) as representative CW agents. CW agent challenges to the Photovac MicroFID Handheld Flame Ionization Detector show that the MicroFID responds only at very high concentrations. In the authors' opinion, this will not provide sufficient warning for the safety of first responders.

The unpredictable CW agent detection performance prevented the establishment of a reliable response curve. The results produced from the minimum detectable levels (MDLs) and response factors (RFs) suggest that the MicroFID in its current configuration cannot be used for CW agent detection. The detectors were not sensitive enough to detect CW agents at concentrations within an order of magnitude of the JSOR (Joint Service Operational Requirement) levels for any of the conditions tested.

The evaluation was terminated after the ambient temperature agent sensitivity testing because of the erratic and less than desirable responses to CW agent at ambient temperatures. Further testing of the detectors at other temperatures and other planned tests were considered to be of no value.

APPENDIX

Domestic Preparedness Test Plan 1998

Subject: Project Domestic Preparedness: CBIAC Task 252
Chemical Warfare Agents Detection Evaluation of Detection Devices.

1. Background

U.S. Army CBDCOM has tasked Battelle to conduct a study to identify the detection equipment that local emergency responders would require for responding to a terrorist incident involving a chemical warfare (CW) agent. The following detection devices were identified and are currently accessible by the HAZMAT Responders in Baltimore County, MD and the Maryland Department of the Environment are:

- HNU Model PI-101
- Photovac MicroFID
- OVA PID(Photo Ionization Detector)
- Mine Safety Appliances Passport (FID)

The study also revealed suggestions from Draeger Safety Incorporated on the availability of two drafted kit configurations of commercial tubes for possible use in CWA detection. These kits are designed to detect thio-ether, organically based nitrogen compounds, phosgene, cyanide, organic arsenic compounds, arsine, and phosphoric acid ester.

Detectors received were:

- HNU Model PI-101
- Foxboro TVA 1000B PID+FID Dual
- Mine Safety Passport (PID)
- MiniRAE (PID), supplied by RAE Systems
- Colorimetric Draeger Tubes

2. Purpose of Test

The purpose of this test is to assess the operational characteristics, the instrument's sensitivity to chemical agents (GB, GA and HD) under different concentrations to determine their respective response factors. All of the detectors were to be calibrated with a reference calibration gas. The value display on the respective panel or meter is calculated into PPM (parts per million) readings corresponding to the calibration. Response factor, when determined by corresponding PPM reading when challenged to a known sample concentration, is required to correlate the actual PPM of the suspected gas detection (i.e., if the type of gas being monitored is known).

Additional temperature and relative humidity extreme tests, to the extent possible, will be conducted. Responses to potential interference substances, especially in smoky environments will be collected.

3. Tasks to be performed

a. Operating Characteristics: The characteristics as to the start up and the time required for achieving readiness for reliable operation and stability.

b. Agent Sensitivity: The detectors will be challenged with GA, GB, and HD agents using three different concentrations to determine their respective threshold sensitivity and response factor (RF) using agent vapor generated with zero air at ambient temperature and 0% RH. The threshold concentration is the concentration at which an alarm will occur within 2 minutes of exposure time. Because these detectors are not set for these agents, the 2-minute response PPM values will be recorded to provide a response curve for each agent and detector.

The determined threshold concentration level (common to the candidates) will be used for subsequent tests at different environment conditions. The Draeger kits, expected to have significant higher sensitivities, will require a separate set of concentration levels.

c. Relative Humidity Effects: Effect of RH extremes at ambient temperature versus detection sensitivity using the determined threshold concentration will also be conducted. While at that concentration, the highest relative humidity of the agent vapor atmosphere will be tested up to 90%RH and plotted against the response curve to observe the effect.

d. Field Interference Test: The unit will be field tested with common potential interference substances such as engine exhausts, burning fuels, and other burning materials to include common clothing and building materials.

These detectors will be challenged using vapor from gasoline, diesel, anti-freeze and AFFF (Fire Fighting Foam). They will be tested in a laboratory controlled generation system where the relative concentration (up to 5%) can be semi-quantified.

The ability for these detectors to maintain the CW agent detection capability in a smoky environment is a major concern. In the event that a smoky environment causes no response from these detectors during the field test, such a smoke will be (attempted to) generated in the laboratory to mix with a CW agent vapor to demonstrate whether the CW agent detection ability remains.

e. Operation/Sensitivity at Temperature Extremes: Operation at temperature extremes of -20 and +45°C, not to exceed the parameter of the detectors, will be conducted to observe the operational characteristics. Threshold level of agent GB, GA and HD responses at the temperature extremes using no RH will be compared to the response curve obtained at ambient condition.

4. Stability and reliability

Observations made during the test program will be used to determine the relative stability and reliability of the devices. Abnormalities will be observed. Due to the unknown life expectancy of these detectors, no additional stability runs are planned at present.

5. Remarks

The scope of this test program suggests that statistical error analysis is not deemed necessary. Because each test point will be conducted with repetitions (3) and that the test is conducted in a paralleled manner, the comparative data collected will give an insight to the characteristics of these units' performances.

Extent of this evaluation is limited to the above mentioned characterization criteria. Other tests such as vibration, EMI, and temperature/humidity storage ability need not be considered at this time until these test items have demonstrated successful CW agent detection performances.

Test conditions will not exceed the items' potential. These detectors will not be subjected to conditions exceeding their specifications. This evaluation is intended to develop a database of CW agent detection capability of the existing detection devices that are available to the HAZMAT Responders. Testing criteria and procedures may be modified when necessary to gain the appropriate information from this evaluation.

Results from this evaluation will serve as a basis for further consideration in determining the relative preparedness of the HAZMAT Responders for CW agent detection.

6. Material Required

Two each of the above mentioned detectors with accessories and sufficient Draeger Kits with appropriate tubes. It is estimated to allow a minimum of 10 tubes per condition to allow replications, potential waste, and blank testing.

Conditions: Each detector will be exposed to the following conditions for each CW agent for sensitivity determination.

- Threshold detection limit at ambient temperature, low RH
- Threshold detection limit at ambient temperature, medium RH
- Threshold detection limit at ambient temperature, high RH
- Threshold detection limit at low temperature, Zero humidity
- Threshold detection limit at high temperature, low RH
- Threshold detection limit at ambient temperature, low RH, with (AFFF or Diesel fume?)
- Threshold detection limit at ambient temperature, low RH, with smoke interference
- Array of potential field interference materials (to be determined).

There are seven CW agent sensitivity conditions per agent. Testing with three agents (GB, GA, and HD) will total to 21 conditions=210 tubes. The array of potential field interference materials will determine the additional tubes required (allow five tubes per material).

7. Point of contact: Kwok Ong, SCBRD-ENM-S, X5-8560.